

LABNOTES

Fall 1997



The Newsletter of the Wisconsin Laboratory Certification and Registration Program
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DNR seeks approval for public hearings on amendments to NR 149

The Wisconsin Department of Natural Resources (DNR) is seeking approval to hold public hearings on proposed amendments to chapter NR 149, Wis. Adm. Code, at the October 21 - 22, 1997 Natural Resources Board Meeting in Madison, WI. The proposed amendments are intended to clarify several sections of the code pertaining to reference samples, applications and the annual renewal process. The proposed changes include a measure that will create two separate base fees; one for certified labs and one for registered labs. Another item included in the proposal is the creation of a test category for immunoassay testing. New tests for explosives by liquid chromatography and glycols by gas chromatography will also be added. If the Board authorizes the changes for hearing, the Department will hold public hearings and accept oral comments on the amendments in early December 1997. The period for submitting written comments will run from October 23 through December 20. After the comment period, the Department will revise the rule proposal based upon the public comments received and resubmit the rule to the Board for final authorization, probably in February 1998. Once the amendments are approved by the Board, they will go to the appropriate legislative committee for review and approval.

The Department hopes to have the amendments in place prior to July 1, 1998.

Copies of the proposed amendments and supporting documentation will be available on the DNR's web site as soon as they are approved for hearing by the Board. If you do not have access to the internet and would like to receive a paper copy of the proposed amendments, please contact Jeff Ripp at (608) 267-0579 or by e-mail at rippj@dnr.state.wi.us.

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The information presented in this newsletter is intended as guidance for certified and registered laboratories. This newsletter does not establish new policy or supersede existing Department policies or procedures. This newsletter does not limit the Department's authority established under s. 299.11, Wis. Stats.

EPA pulp and paper methods fit into existing test categories

The DNR's Bureau of Watershed Management has requested that several pulp and paper mills in Wisconsin monitor their effluent using the analytical procedures found in the 1993 *Analytical Methods for the Determination of Pollutants in Pulp and Paper Industry Wastewater* [USEPA 821-R-93-017]. The Department has not previously certified or registered laboratories to perform these specific tests which include: 1613A (dioxin), 1650 (adsorbable organic halides), 1653 (chlorinated phenolics) and NCASI 253 (color). However, these procedures are similar to those that laboratories are currently performing for other regulatory programs (e.g., municipal wastewater). Laboratories that wish to perform these pulp and paper tests for compliance monitoring in Wisconsin must be certified for the appropriate test category. For example, laboratories need to be certified in Category 17 for 1613A, Category 12 for 1653, Category 7 for 1650 and Category 5 for NCASI 253. If your laboratory is currently certified or registered for the appropriate test category and would like to bring the pulp and paper methods online, please submit a brief letter notifying the Department that you intend to add these methods to your analytical repertoire. Your laboratory must complete the initial demonstrations of precision and accuracy and detection limit studies prior to submitting compliance data. If you fail to notify the Department of your intent to perform these methods, your laboratory will not appear on any laboratory lists that we publish. For more information, contact Jeff Ripp at (608) 267-0579 or by e-mail at rippj@dnr.state.wi.us.

Wisconsin certification calendar changes

Some of you may have noticed that your new certificate is valid until August 31, 1998. The DNR hopes that this change will improve the service we can provide to our labs. The DNR will continue to bill labs and collect reference samples (PE samples) in June and July, but now we will be able to print a new certificate for your laboratory before the old one expires. All laboratories will be moved onto the September 1 through August 31 renewal cycle, including DMR labs that have traditionally been on a calendar year cycle. This change will not affect your laboratory's billing or reference sample arrangement. If

you have any questions about the new certification period, please contact Mike Kvitrud at (608) 261-8459 or by e-mail at kvitrm@dnr.state.wi.us.

Special note for those labs whose certificate ends on December 31, 1997: When the results of DMR-QA 17 are available in January, we will issue new certificates ending on August 31, 1998. Because of the shortened period of the certificate, the Department will not require that you analyze another set of reference samples before August 31, 1998. You will receive a new certificate in August.

SW-846 Update III released, effective date clarified

EPA released Update III to SW-846, *Test Methods for Evaluating Solid and Hazardous Waste* in August of this year. This update contains many new procedures and technologies and deletes several outdated ones. In the preamble to the final federal rule [62 FR 32452], EPA stated that the update would become effective immediately. EPA has since backed away from this position, allowing labs more time and flexibility in implementing the new procedures. EPA is asking that states implementing the Resource Conservation and Recovery Act (RCRA) programs allow laboratories up to six months (until March 1998) to implement the new SW-846 procedures.

The Wisconsin Laboratory Certification Program is incorporating Update III by ref-

erence in the amendments to chapter NR 149, Wis. Adm. Code (see page 1). These amendments are anticipated to become effective in July, 1998. Laboratories that use the SW-846 methods should begin updating their method detection limit and initial demonstration of capability information and revise their standard operating procedures as soon as possible, but no later than the effective date of the NR 149 changes. Laboratories that are ready to use the update before the amendments to Chapter NR 149 are promulgated will be allowed to do so provided they have met all of the method performance criteria. If you have any questions, please contact Alfredo Sotomayor at (608) 266-9257 or by e-mail at sotoma@dnr.state.wi.us.

Wisconsin DNR Laboratory Certification Web Site Has Moved!!

Please note that the Wisconsin DNR Laboratory Certification Program web site has moved as result of the recent reorganizational changes in the agency. The site has also been re-designed with a new look. The program's web site is now located at www.dnr.state.wi.us/org/es/science/lc.

Standards approved in NELAC III

The Third Annual National Environmental Laboratory Accreditation Conference (NELAC III) took place in Dallas on July 29 - 31. In the relative comfort of the Wyndham Anatole's controlled environment, sheltered from the Texas 99° heat, Federal and State regulators, and other interested parties met and decided the fate of a national environmental certification program.

The most significant outcome of NELAC III was that all proposed accreditation standards were approved by the Conference's two houses, thus making them official. Although there are selected sections in some standards that are still reserved or incomplete, their current status would enable prospective accrediting authorities to use the approved standards to have a viable program.

Nine states; Minnesota, Illinois, Hawaii, Washington, Florida, New York, Utah, Connecticut and Virginia, intend to request NELAP recognition as soon as it becomes available, which is projected to be June 1998. Once these states become accrediting authorities, they will be ready to accept applications, visit, and accredit laboratories. If it all goes according to plan, by late 1998 or early 1999 there will be laboratories in the United States bearing the NELAC logo in their certificates. To avoid the potential problems of having to set priorities for granting NELAP recognition and NELAC accreditation, all accrediting authorities and laboratories will be granted approvals in blocks, at the same time. After this first round, application dates will decide the order for on-site visits and approvals.

The ease with which the standards were approved surprised many of the conference's participants. Many felt that it was important to have a set of rules, however imperfect, that could be the basis for creating an unofficial pilot. EPA, for its part, was under some pressure to deliver some tangible proof to justify the massive efforts undertaken by so many. At the end of the conference, even those with substantial reservations felt that much had been accomplished.

Page 5 contains some of the conference highlights and a few of the revisions made to the standards during NELAC III. In this manner, the standards are likely to be the principal content of the statutes and administrative codes of accrediting authorities.

NELAC: Talk the Talk

It is essential that you become familiar with the NELAC fundamental lexicon before you take a stroll in the partially charted territory of national environmental laboratory accreditation.

Accreditation: This is the NELAC term for certification. The term certification is reserved for analysts, but the current standards do not require analyst certification.

Accrediting Authority: The government body from which a laboratory can obtain accreditation.

Assessment: Equivalent to audit, usually accompanied by the words "on-site", as in our usage of "on-site evaluation" or "on-site audit".

Assessor: The new and improved term for auditor, now devoid of its former fiscal associations.

Contributor: The term used to describe non-government conference participants. Contributors can, of course, contribute and be committee members, but may not vote.

ELAB: An acronym for the Environmental Laboratory Advisory Board. This board advises EPA and NELAC on environmental policy matters and makes recommendations to both bodies.

IDAC: An acronym for initial demonstration of analytical competence; equivalent to our IDC.

Highlights of NELAC III

NELAP: The National Environmental Lab Accreditation Program. NELAP ensures implementation of the standards. Unlike NELAC, NELAP has some regulatory authority.

Primary Accrediting Authority: The State where a lab is located or that is responsible for assessing the lab.

Proficiency Testing (PT) Samples: The same as performance evaluation samples or what we call reference samples.

PTOB: Proficiency test oversight body, an organization independent from EPA that will oversee and approve multiple providers of PT samples.

Recognition: What NELAP grants to prospective accrediting authorities that meet the NELAC standards.

Responsible Party of Record: Refers to the Laboratory Director or the person who has authority, as in power of signature, to make his or her laboratory comply with the NELAC standards.

Secondary Accrediting Authority: Government bodies that grant accreditation to a lab based on the work that another body (the primary accrediting authority) has invested in verifying adherence to the standards. Essentially, they grant reciprocal accreditation to labs.

Standard(s): A series of protocols that will have the weight of regulations as accrediting authorities adopt the standards into their regulations.

- A body independent from NELAP, at this point identified as NIST, will be in charge of approving reference sample providers.
- Auditors currently employed will have up to five years to take and pass NELAP-approved training courses.
- Laboratories have the right to seek exclusion from the public record of certain types of information by requesting a "confidential business information" determination from the accrediting authority.
- The evaluation of health, safety, and chemical disposal practices of laboratories were removed from the components of assessments.
- A draft of an assessors' manual was made available for comment. Checklists for assessors have not been devised.
- Interim accreditation is still available for laboratories that have met all accreditation requirements but have not been visited on-site by an accrediting authority.
- The Conference could not agree on the qualifications and credentials of a laboratory's responsible party of record.
- Substantive changes were not made to the Quality Systems standard. Still under debate are alternatives to the MDL for estimators of sensitivity and improved calibration requirements.
- The place of performance based measurement systems (PBMS) in the Quality Systems standards was discussed, but an informational appendix dealing with it was not presented for adoption.
- The teams performing assessments of the accrediting authorities will not include private contractors or any other third party. EPA and the States felt that assessing accrediting authorities was an inherent governmental function.



DNR accepting nominations for the Lab of the Year

The Wisconsin Department of Natural Resources annually recognizes two registered laboratories for their outstanding commitment to producing high quality data. The awards are presented at the Natural Resources Board Meeting in March of each year. The DNR presented the 1997 awards to WP&L - Edgewater and the city of Medford Wastewater Treatment Plant. Last year, we received nominations for a number of extremely qualified facilities, and choosing the two recipients was difficult. We hope to continue this trend for 1998.

The DNR is accepting nominations for the 1998 awards until December 31, 1997. There are two award categories: one for smaller facilities and one for larger facilities. Small facilities generally test a small number of samples each year and are registered only in categories 1 - 4. Larger laboratories are registered in more test categories than just 1 - 4 or analyze a large number of samples each year. Nominations are open to DNR staff and to the public, but a laboratory may not nominate themselves for the award. The winners will be selected by a diverse committee in January. Nominees for the award must meet the following criteria:

- The lab must be a Wisconsin registered laboratory in good standing, with no outstanding enforcement actions. (Certified laboratories will not be considered.)
- Nominees must be located in the State of Wisconsin.
- Nomination forms must be received by December 31, 1997.

To nominate a Wisconsin registered laboratory for the 1998 Lab of the Year award, simply complete a nomination form and attach a brief summary no more than three pages long of why you think the labo-

ratory deserves the award. Be sure that you can clearly justify, with specific examples, which of the criteria listed on the form you feel that the nominee meets. Nomination forms are available from Jeff Ripp at (608) 267-0579. Alternatively, nomination forms and instructions can be found on the DNR's web site. Please return completed forms no later than December 31, 1997 to the Wisconsin Department of Natural Resources, c/o John R. Sullivan- SS/6, 101 S. Webster St., P.O. Box 7921, Madison, WI 53707-7921 or by FAX at (608) 266-5226.

EPA to 'externalize' reference samples in 1998

As announced in previous issues of *LabNotes*, EPA is getting out of the reference sample business. The last studies EPA will offer are DMRQA18, WP040 and WS041, all scheduled for mid-1998. The externalized EPA programs will have minimal effect on the operation of the Wisconsin Laboratory Certification and Registration Program because Wisconsin already accepts results from a number of alternate providers. However, laboratories need to prepare for the day when they will no longer receive free samples from EPA. To avoid possible delays during certification renewal for FY 1999, laboratories need to make arrangements with one of the alternate providers listed on page 7. Laboratories certified for Safe Drinking Water tests must still analyze EPA's WS samples until the final study (WS041).

Please see 'Externalization' on page 7

'Externalization' from page 6

After WS041, the Department will specify alternative sources for drinking water samples. If you wish to be removed from the DMR-QA or WP nomination list before the final studies, please contact Mike Kvitrud by

phone at (608) 261-8459 or by email at kvitrm@dnr.state.wi.us. For more information on reference samples and providers, see section 3 of the *Program Information and Requirements* booklet (the "Yellow Book") [WDNR-PUBL-TS-007].

Reference Sample Source	Phone Number
Analytical Products Group (APG)	(800) 272-4442
Analytical Standards Inc. (ASI)	(800) 283-4844
Environmental Resource Associates (ERA)	(800) 372-0122
New York Department of Health	(518) 474-8519
Wisconsin State Laboratory of Hygiene (SLH)	(608) 833-1770 ext.107

DNR to request laboratory detection limits

In 1995, the Natural Resources Board approved amendments to chapter NR 149, Wis. Adm. Code, requiring laboratories to report data down to their statistically determined detection limit for a select list of substances with health-based standards at or near the analytical detection limit. Further, the Board mandated that the Department publish a list of substances meeting that criteria each year, and that the list reflect any changes in the standards. To follow up with this mandate, the Department will be requesting detection limit information from laboratories that are certified or registered for any of the chemicals in the *'Substances of Concern'* list on page 8. If you receive the request, you should submit your limit of detection (LOD) and limit of quantitation (LOQ) information for the specified substances in a water matrix. The request form should not take long to complete and your

laboratory should not have to run new LOD/LOQ studies if your lab's current studies are still valid.

This information is being requested to help both the Department and the public better understand the range and variability of LODs and LOQs being reported by the laboratory community and to determine the level of compliance with the low-level data reporting requirement. Once all of the necessary information is received, the Department will publish a paper giving statistical summaries for each analyte and method. The final report will be available to the public. Since this is an important study, please be sure to completely fill in all of the requested information. No laboratories will be identified or associated with their results on the final report. Questions about the study should be addressed to Mike Kvitrud, (608) 261-8459 or by e-mail at kvitrm@dnr.state.wi.us.

Substances of Concern at Low Levels

INORGANICS

Metals

Antimony
Beryllium
Cadmium
Lead
Thallium
Mercury
Chromium (Hexavalent)

ORGANICS

Acids/Phenols

Pentachlorophenol (PCP)

Benzidines

Benzidine

Haloethers

Bis(chloromethyl)ether

Nitroaromatics

2,4-Dinitrotoluene
2,6-Dinitrotoluene

ORGANICS

Polynuclear Aromatic

Hydrocarbons

Benzo(a)pyrene

Phthalates & Adipates

Di(2-ethylhexyl)phthalate

Nonpurgeable Chlorinated

Hydrocarbons

Hexachlorobenzene

Dioxins/Furans

Dioxin

PCBs

Polychlorinated biphenyls

Chlorinated Pesticides

DDT and Metabolites
Heptachlor
Heptachlor epoxide
Lindane
Toxaphene

ORGANICS

Carbamate Pesticides

Aldicarb

Nitrogen Pesticides

Alachlor
Dimethoate
Parathion
Trifluralin

Volatiles

1,1,2,2-Tetrachloroethane
1,1,2-Trichloroethane
1,3-Dichloropropene
(cis/trans)
Bromodichloromethane
Bromoform
Bromomethane
Chloroform
Chloromethane
Methyl tert-butyl ether
(MTBE)
Methylene Chloride
Vinyl Chloride
Dibromochloropropane
(DBCP)
Ethylene dibromide (EDB)

Did you know...?

The Wisconsin DNR certifies or registers 550 laboratories in 27 different states. In 1996, the Wisconsin Laboratory Certification and Registration program processed 93 applications for certification or registration, including 16 new applications, 66 revised applications and 11 applications for transfer of ownership. The program is divided into two parts; the Central Office in Madison and five Regional Offices statewide. The Central Office is responsible for certifying commercial and safe drinking water labs while the Regional Offices are primarily responsible for registering municipal wastewater treatment and industrial facilities.

EPA announces performance based measurement initiative

EPA released its plans to implement a performance based measurement system (PBMS) for all of its environmental monitoring programs in the October 6, 1997 Federal Register [FR-5903-2]. The agency defines PBMS as a set of processes that specify the data quality needs, mandates and limitations for a the project. These needs serve as the criteria for selecting the appropriate analytical technique for a particular project. Where PBMS is implemented, the regulated community will be allowed to select any appropriate analytical method for use in complying with EPA's regulations. Under PBMS, EPA will establish performance criteria for characteristics such as method precision and accuracy. Laboratories demonstrating that they can meet these criteria would be allowed to modify or choose methods that will suit their needs. The intent of the initiative is to improve overall data quality while encouraging the advancement of emerging analytical technologies. EPA hopes that this will result in less costly monitoring approaches.

Historically, EPA programs have required specific analytical methods to be used by the regulated community. This was frustrating for many laboratories because they had to maintain separate analytical protocols for drinking water, wastewater and solid and hazardous waste samples. The requirement to use specific analytical methods for compliance purposes was one way to ensure a minimum level of consistency and reliability. In some cases, EPA has provided flexibility to use alternate methods for compliance

(e.g., the Alternate Test Procedure process). These alternatives are not very flexible, and are often time consuming and expensive for a laboratory proposing an alternate procedure. Although PBMS appears to be a promising alternative to using the outdated procedures presently required in many programs, EPA has not announced a strategy for uniting all of its various programs under a consistent PBMS context. In fact, EPA intends to implement PBMS on a program-specific basis. This means that EPA may implement PBMS for Safe Drinking Water Act testing before Clean Water Act testing, or vice-versa. Each of EPA's programs is developing an implementation plan. Each implementation plan is supposed to address the specifics of how PBMS will work in specific regulatory programs.

The Notice of Intent published in the Federal Register does not create any regulatory requirements. EPA anticipates proposing amendments to its regulations as necessary to incorporate PBMS into its regulatory programs. EPA is accepting comments on the general nature of the proposal until November 5, 1997. Specific questions EPA would like answered in the public comment process are listed in the Federal Register. Additionally, draft generic checklists describing the documentation for initial and continuing demonstrations of method performance are available at EPA's web site at www.epa.gov/pbms. For more information, contact Carol Finch, Executive Director of the Environmental Monitoring and Management Council, US EPA, (202) 564-6638.

Representative reference samples for pesticide tests

Laboratories certified or registered for pesticide work are required to analyze a representative reference sample for each pesticide subcategory. But what exactly does the phrase 'representative analytes within each subcategory' mean? In the past, laboratories have been required to submit proficiency testing results for a large number of analytes that fall into a particular class. As a result, many labs commented that they were analyzing compounds that are not part of routine monitoring in Wisconsin. This became expensive for laboratories because they needed to buy standards for the sole purpose of analyzing reference samples. Further, many of these analytes are rare enough that they do not give a fair evaluation of capability on routine tests.

To correct this problem, the DNR, in cooperation with the Department of Agriculture Trade and Consumer Protection (DATCP), has evaluated groundwater, surface water and pesticide application data to determine which pesticides are commonly used, found or otherwise important in Wisconsin. Based on this evaluation, the DNR has developed a reduced analyte list of 'key pesticides' that are of special concern in Wisconsin (see the list on page 11). The purpose of the list is to match the certification process to actual chemicals found in the field. The DNR is recommending that at a minimum, reference sample providers include these analytes in their samples. Under the new guidelines, laboratories that analyze pesticide samples will only be responsible for submitting results for these compounds, and not for the entire universe of pesticide chemicals.

Please see 'Reference Samples' on page 11

COUNCIL CORNER

The one thing that we can continue to count on in the laboratory business is change. The National Environmental Laboratory Accreditation Program (NELAP) will certainly offer that. NELAP is essentially a set of federal guidelines that would be implemented on a State level. There has been detailed discussion regarding this program, and several conferences (NELAC) held with stakeholders over the past several years. For a long time it seemed as though there was a lot of wheels spinning, trying to find the right combination of guidelines to have a program that the various parties could embrace. Well, they finally got their arms around it, and it is now progressing quickly. The first group of states will be considered for NELAP in 1998. There are nine states in this group, including our neighbors Illinois and Minnesota.

DNR's staff has been involved in NELAC through all of its growing pains. They have briefed the Council as well as various lab groups along the way. It is now time to look at how Wisconsin's program might dovetail with the National program and what changes would need to take place to allow the adoption of NELAP. The DNR has not stated an official position on adopting NELAP. Various parts of Wisconsin's program would require modification. In light of this, all labs operating under Ch. NR 149, are encouraged to offer input. Your comments, questions and concerns are encouraged and can be voiced through the various representatives on the Council or directly to the DNR staff. You are encouraged to be involved in this process.

Ed. Note: The Council Corner is written and submitted by the Certification Standards Review Council. Ms. Mary Christie is the current Council chair.

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Reference samples for each subcategory must still contain a representative number of analytes. For example, the required number of analytes for each subcategory under Category 14 are: 5 nitrogen pesticides (out of 10 possibilities), 5 triazine pesticides (out of 6 possibilities) and 5 organophosphorus pesticides (out of 6 possibilities).

Currently, the State Laboratory of Hygiene is the only approved reference sample provider that has adopted the guidelines for its proficiency testing program. Other Wis-

consin approved providers are expected to meet the guidelines for at least some subclasses of pesticides. Laboratories should verify that their preferred provider will meet the Wisconsin requirements before submitting results. Keep in mind that providers other than the State Laboratory of Hygiene may include a wider range of possibilities and a greater number of chemicals in a study. For more information about pesticide reference samples, contact Mike Kvitrud at (608) 261-8459 or by e-mail at kvitrm@dnr.state.wi.us.

'Key Analytes' for Category 14 Pesticide Reference Samples			
Subcategory:	Nitrogen Pesticides	Triazines Only	Organophosphorus
# Analytes:	At least 5 (10 Possible)	At Least 5 (6 Possible)	At Least 5 (6 Possible)
Possibilities:	alachlor butylate EPTC (eptam) metolachlor metribuzin trifluralin atrazine cyanazine prometon simazine	atrazine desethylatrazine deisopropylatrazine cyanazine prometon simazine	chlorpyrifos diazinon dimethoate fonofos phorate terbuphos

Update to solid waste table includes CAS numbers

Back in July, 1996 the Department issued revised requirements for reporting environmental monitoring results for solid and hazardous waste samples. A letter dated July 30, 1996 was sent to both groundwater monitoring contacts for solid and hazardous waste facilities, and to Wisconsin certified laboratories reporting solid and hazardous waste data. Attachment 3 to that letter was a waste management parameter table sorted by parameter name and by parameter number.

By request, the DNR has added a number of new parameters to this table. The Department has also added Chemical Abstract Service (CAS) numbers for many parameters and produced a different permutation of the table with the analytes sorted by CAS number. CAS numbers are unique identifiers for chemicals, much like social security numbers are for people. Many laboratories

See 'Solid Waste' on page 12

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use CAS numbers in their computer systems for managing testing data. The new version of the table should make it easier to find parameter numbers for chemicals that have more than one name. You can obtain the updated table electronically by downloading it from: 1) the DNR Remediation and Redevelopment and Waste Management computer bulletin board, or 2) the DNR world wide web site on the internet. Or, you can request a paper copy of the table by calling the groundwater data file manager at (608) 267-0546 or (608) 267-7550.

To access the bulletin board, dial (608) 261-6455 with a computer telecommunications program (such as Hyperterminal for Windows 95, Procomm or Telix) and a modem (8-N-1). The terminal setting should be "ansi bbs" and the transfer protocol should be "Zmodem". When you get into the bulletin board, look in Directory 5 for the file named PARMRPT.ZIP and download it. You'll need unzip software to extract the file downloaded from our bulletin board.

To access the file from DNR web site, point your web browser to www.dnr.state.wi.us/org/aw/rr/errhw, find the file named PARMRPT.EXE and download it. To extract the file downloaded from our web site, you'll only need to: 1) get to a command prompt for the directory to which you downloaded the file, type the downloaded file name and press Enter, or 2) double-click on the downloaded file name in Windows File Manager, Windows 95 or NT Explorer, or your web browser.

If you have a problem accessing either the DNR bulletin board or the web site please contact the bulletin board system manager, Chris Zenchenko at (608) 267-3543.

WPDES wastewater permits, SLH reference samples are not the same as certification

Recently, the Department has expanded WPDES requirements for total phosphorus to facilities outside of the Great Lakes Basin. As a result, many wastewater labs have begun measuring total phosphorus in their effluents. In a few cases, the facilities have analyzed and passed reference samples from the State Laboratory of Hygiene and assumed that this meant that they were certified or registered by the DNR. This is absolutely NOT true; having total phosphorus in your WPDES permit and passing a reference sample does not automatically grant your lab total phosphorus on your Wisconsin laboratory (NR 149) certificate. The proficiency testing program administered by the State Laboratory of Hygiene and the DNR's Laboratory Certification program are separate entities. Although the DNR uses the State Lab's samples for certification and registration purposes, these samples in and of themselves do not grant certification.


Please check your chapter NR 149 certificate to make sure that you are registered or certified for total phosphorus if you are reporting results for this analyte on your Discharge Monitoring Report (DMR) (Form #3200-28). If you are analyzing and reporting total phosphorus on your DMR but it is not on your laboratory certificate, your facility must file a revised application to have total phosphorus added to your certificate. Application forms are available for downloading on the DNR's web site or by calling John Condron at (608) 267-2300 or by e-mail at condrj@dnr.state.wi.us.

Update on Low-level Mercury Laboratories

The Spring 1997 *LabNotes* highlighted the Department's mercury strategy and the move to obtain higher quality data by using more sensitive analytical procedures. At that time, only one laboratory had been recognized by the DNR to perform low-level mercury analysis. The Department now recognizes three commercial laboratories and two public laboratories that can achieve the required 20 ng/L detection limit in wastewater. These labs are:

Northern Lake Services, Crandon, WI
S-F Analytical, Milwaukee, WI
Frontier Geoscience, Seattle, WA
WI State Laboratory of Hygiene, WI
Madison Met. Sewerage District, WI

The DNR is sending out this list of laboratories with the wastewater permit application packages. The wastewater program will only accept low-level mercury from laboratories that are on this list. Laboratories certified or registered for traditional mercury analysis can continue to submit data for non low-level projects. If you are interested in having your laboratory's low-level mercury capabilities recognized, you need to demonstrate that (1) your laboratory can achieve a sensitivity of 20 to 50 ng/L in wastewater, (2) ambient mercury contamination in the lab has been controlled and (3) your lab can meet the method specified quality criteria. You may contact Donalea Dinsmore at 266-8948 or by e-mail at dinsmd@dnr.state.wi.us if you have questions about applying for low-level mercury certification.



Reduced Nitrogen Pesticides in EPA Water Supply PE Study

One notable change in this latest EPA WS study (#39) was the lack of Nitrogen Pesticides. prometon, metribuzin, bromacil, metolachlor and butachlor were not included, leaving only alachlor, atrazine and simazine. Traditionally, the WS study has been accepted for granting or renewing non-drinking water nitrogen pesticides as well as the SDWA (drinking water) nitrogen pesticides. That did not change for the EPA's WS #39. However, if future EPA WS studies (only #40 and #41 remain) do not contain five or more nitrogen pesticides, then the studies may not be considered acceptable for non-drinking water nitrogen pesticide renewal (see article on page 10). Labs which already analyze nitrogen pesticide reference samples from another provider (SLH, APG, ERA) will not be affected. Also, this will not affect a lab's SDWA nitrogen pesticide certifications. If you need more information, please contact Mike Kvitrud at (608) 261-8459 or by email at kvitrm@dnr.state.wi.us.

The Auditor's Corner

Alfredo Sotomayor, Senior Audit Chemist

CONFIRMATION: PART III-PARALLEL LIVES

Many laboratories habitually provide simultaneous dual column analysis for many GC determinations. This is most common for analyses of pesticides and PCBs, following a practice initiated by the EPA's Contract Laboratory Program (CLP) more than a decade ago. This is less common for

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other organic analytes because the CLP has always required GC/MS analyses for volatiles and semi-volatiles. In these systems, two GC columns are connected in parallel to two detectors and when analytes are detected in both columns, one is used to give a numerical result, quantitative or qualified, while the other is used to provide the confirmatory analyses. Not many guidelines for reporting results from these dual systems are available. In this column, going where angels fear to tread. I will suggest some.

First Principles

- Start with two columns that are approximately equal in sensitivity, but with sufficiently different polarities.
- Calibrate both columns fully. This is not that difficult for dual column split injection systems because each column gets a dose of each standard and sample injected.
- Perform an MDL study and reduce the corresponding data from both columns. Calculate an MDL for each one of them.

In the best possible systems, both columns would be equally capable of providing quantitative results. In reality, some columns are more sensitive for selected compounds and some may not be able to resolve all analytes needed to be reported in a run. Therefore the sensitivity and the selectivity of each column must be considered in evaluating the validity of the system.

Sensitivity

How similar must the sensitivity of the columns be? No regulatory guidelines are available, but I suggest that the **least** sensitive column's MDL (or LOD) for a specific

analyte should not exceed the other columns LOQ for the same analyte. The farther apart these two quantities are, the more questionable the confirmatory capacity of the dual column system becomes. The most sensitive column should be able to detect analytes at or below the regulatory levels.

Selectivity

Ideally all reported analytes would be resolved by each of the columns, but this seldom happens. Both columns combined should resolve all reported analytes in a run and I suggest that each in turn should resolve about 90% of the reported analytes. The coeluting 10% should consist of pairs--a coeluting triplet is a mob in chromatography. The same compounds could coelute in both columns, but as long as the coeluting **pairs** are dissimilar, confirmation and quantitation may still be possible.

If more than 10% of the reported compounds coelute, then it is time to optimize run conditions, change columns, or seriously consider GC/MS for confirmation. Is this 90% criterion required by any rule? Only by the rule that guides you toward achieving excellence.

It is imperative that retention time windows be established and checked properly for these systems.

Quantitation Options

When the two columns produce equally defensible results, the primary column is the column used to report numerical results while the secondary column becomes the one used for confirmation. Ideally, **the most sensitive and selective column should be chosen as the primary column**. Two options are then available for quantitation:

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Q1 One column is exclusively used as the primary column for all analytes in a run. All numerical results are obtained from one column.

Q2 The primary column is analyte specific and therefore, not the same for all analytes in a run. Each column is used to quantitate and confirm a selected set of different analytes.

The first option (Q1) is categorical and easier to manage, but does not utilize the full potential of the dual column system and may not always be feasible, for instance, when the column fails continuing calibration for a compound. If you are to use the same column for all quantitations, apply the 90% rule once again, this time choosing the column that not only resolves 90% of the analytes, but that provides greater sensitivity for 90% of the reported compounds, or at least those compounds that have low regulatory limits.

The second option (Q2) is more versatile and probably more defensible, but requires declaring **up-front** the reporting scheme that will be used, barring matrix interferences or chromatographic anomalies, to prevent questionable judgments that may lead to censoring.

And of course, no matter what quantitation option you choose, you must consider the rule that your laboratory follows for deciding **when** to report a result.

Decision-Making Rule for Reporting Results

These are three commonly used decision-making rules for reporting results:

R1 Numerical results are only reported when an analyte is detected by both columns.

R2 Numerical results are reported for all analytes detected by either column.

R3 Numerical results are only reported for analytes detected by the primary column.

When analytes are detected by both columns, a laboratory could opt to report both results, but because this is not common practice and because doing this would only involve transmitting acquired information, not evaluating it, I will not discuss it further except to add that in this case, the data user, not the laboratory, could be making the final decision on the results.

The table on page 16 illustrates how these different decision rules affect what a laboratory might report when faced with a set of detected analytes in a simultaneous dual column system. I have made the following assumptions as well:

- Detection means obtaining a valid signal at or above the MDL of an analyte. Detected results below a valid MDL are not reported.
- The laboratory reports all results, with appropriate flags, detected between the LOD (MDL) and the LOQ.
- The primary column is the more sensitive of the two.

The reports suggested by this table are valid when the sensitivity of the two columns are close to equal. If this is not the case, following decision rules **R1** and **R3** may cause problems when the laboratory detects analytes for which we require reporting results down to the MDL. The table

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DECISION RULE COLUMN DETECTED	R1	R2	R3
BOTH	REPORT PRIMARY COLUMN RESULT	REPORT PRIMARY COLUMN RESULT	REPORT PRIMARY COLUMN RESULT
PRIMARY ONLY	N.D. > MDL SECONDARY COLUMN	REPORT RESULT; QUALIFY AS UNCONFIRMED	REPORT RESULT; QUALIFY AS UNCONFIRMED
SECONDARY ONLY; RESULT IS BETWEEN LOD & LOQ OF PRIMARY	N.D. > RESULT OF SECONDARY COLUMN	REPORT RESULT; QUALIFY AS UNCONFIRMED	N.D. > MDL PRIMARY COLUMN
SECONDARY ONLY; RESULT IS ABOVE LOQ OF PRIMARY	N.D. > MDL PRIMARY COLUMN	REPORT RESULT; QUALIFY AS UNCONFIRMED	N.D. > MDL PRIMARY COLUMN

N.D. = Not Detected

also indicates that the reports associated with **R3** are only slightly better than what a laboratory might report by using a single column system.

Finally, note that even when compounds are detected in the two columns you still could be reporting a false positive. "The coincidental presence of two unrelated components in a sample showing up in the right retention time windows on both column approaches certainty as the background in the

sample increases." (Environmental Laboratory Data Evaluation; Berger W., McCarty H., Smith R.; Genium Publishing Corporation, 1996) For environmentally significant analyses, it is still wise to try to confirm by GC/MS. When the sensitivity of the GC/MS is still a limiting factor, injecting more than the customary amount, or using selected ion monitoring (SIM) judiciously can be attempted.

EPA Methods on CD-ROM!

The EPA has released its water methods on CD-ROM. Information on how to order these methods or other documents is available on the Office of Water's web site: www.epa.gov/OW. The SW-846 methods are also available on CD-ROM through the Solutions Software Corporation's web site: www.env-sol.com.

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NEWS BRIEFS

Pollution Prevention: Laboratories interested in reducing the amount of waste they generate should contact the DNR's pollution prevention specialist Kim McCutcheon. Kim is the sector specialist for both environmental and health laboratories, and is particularly focused on reducing mercury waste. Kim's phone number is (608) 267-0876.

PECFA: Laboratories and consultants dealing with contaminated soils from underground storage tanks may be interested in contacting Miles Mickelson, in the Department of Commerce. Miles is a good source of information about PECFA work and reimbursement. Miles' phone number is (608) 267-4545.

SDWA: EPA is requesting public comments on a proposed list of compounds in drinking water that may require regulation under the 1996 Safe Drinking Water Act. Under this Act, EPA has to reevaluate potential compounds every five years for possible inclusion and regulation. The October 6, 1997 Federal Register lists 58 chemicals and 13 microbiological that EPA is considering. Comments on the list are due by December 5, 1997. For more information, call the Safe Drinking Water Hotline at 1-(800) 426-4791.

Wisconsin Laboratory Certification Program Web Site ***www.dnr.state.wi.us/org/es/science/lc/***



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